DERWENT-ACC-NO: 1995-085710
DERWENT-WEEK: 199512

TITLE: Extn. of sugar cane wax compsn. from inexpensive raw material - by heating mixt. of ethanol added to sugar cane residue and recrystallisin g filtered mixt.

PATENT-ASSIGNEE: NISSHIN OIL MILLS LTD[NISW]

PRIORITY-DATA: 1993JP-0177649 (June 25, 1993)

PATENT-FAMILY:

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APPLICATION-DATA:

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INT-CL_(IPC): C11B011/00; C11B013/00

ABSTRACTED-PUB-NO: JP07011284A
BASIC-ABSTRACT: Extn. of a sugar cane wax compsn. comprises adding ethanol to the residue produced in the squeezing process in prodn. of sugar from sugar cane to a final alcohol concn. of at least 50 deg.C, heating the mixt. under elevated pressure to a temp. of at least the b.pt. of ethanol, filtering the solid while the mixt. remains hot and precipitating crystals from the filtrate.

Pref. the pressure is 2-5 kg/cm2. Pref. the mixt. is heated to 100-150 deg.C. Pref. the residue is the filter cake produced in the process of cleaning sugar.
Pref. the ethanol is hydrated and has an ethanol content of 70-90 wt.%.

ADVANTAGE - The method permits extn. from the sugar cane-related inexpensive raw material with a safe single solvent. Though carried out in a hydrated system, it achieves high purity and yield.

In an example, 100 g of a dried prod. of a filter cake of sugar cane having a wax content of 4% and a water content of 20% was put in a high-pressure container provided with a stirrer, added with 800 ml of 90% ethanol, pressurised to 3 kg/cm2 and stirred at 120 deg.C for 2 hrs. The mixt. was filtrated at 80 deg.C and the resultant filtrate was cooled to 25 deg.C and left standing for 5 hrs. to ppte. The resultant crystals were filtered to obtain 3.9 g sugar of a m.pt. of 78-82 deg.C. The sugar was decoloured with 35% H2O2 soln. to obtain a light yellow prod.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS:
EXTRACT SUGAR CANE WAX COMPOSITION INEXPENSIVE RAW MATERIAL HEAT MIXTURE ETHANOL ADD SUGAR CANE RESIDUE RECRYSTALLISATION FILTER MIXTURE

DERWENT-CLASS: D17 D23

CPI-CODES: D06-A; D10-A01;

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JPAB

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TITLE: METHOD FOR EXTRACTING SUGAR CANE WAX COMPOSITION
PUBN-DATE: January 13, 1995
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APPL-NO: JP05177649
APPL-DATE: June 25, 1993
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ABSTRACT:
PURPOSE: To extract the high-purity subject compound in high yield and excellent economic efficiency by adding ethanol to the residue in squeezing process of sugar cane and heating the residue under pressure, filtering the mixture and depositing crystals material from the filtrate.

CONSTITUTION: Ethanol (preferably water-containing alcohol) is added to the residue obtained in squeezing process in producing sugar from cane wax so that alcohol concentration becomes ≥50wt.% ZM, (preferably 70-90wt.%) and the mixture is heated to the boiling point or above (preferably 100-150°C) of ethanol under pressure (preferably 2-5kg/cm<SP>2</SP>) and solid content is filtered in heating and crystals are deposited from the filtrate to extract the
objective composition. Furthermore, the residue is preferably a filter cake obtained in cleaning process of sugar.

PATENT ABSTRACTS OF JAPAN

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INATA JUNICHI

(54) METHOD FOR EXTRACTING SUGAR CANE WAX COMPOSITION

(57)Abstract:

PURPOSE: To extract the high-purity subject compound in high yield and excellent economic efficiency by adding ethanol to the residue in squeezing process of sugar cane and heating the residue under pressure, filtering the mixture and depositing crystals material from the filtrate.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] this invention relates to the extraction technique of ** and a ****** wax constituent.
[0002]
[Description of the Prior Art] ** and a ****** wax are white low-like matter considered to have played the role from which it mainly exists in the **** front face of ** and ******, and **** is protected. In recent years, as a physiological active substance use [higher alcohol, such as a ********** Norian contained in this wax, / for health food or the drug], it begins to manufacture the lubricant for OA equipment, and it attracts attention in various fields.
[0003] In order to manufacture such ** and a ****** wax, the following technique has been adopted conventionally. That is, after gathered a harvest, squeezing a ** and ******, ****ing honeydew, using a ** and float ****** (it is called the so-called bagasse and the so-called filter cake, and many are used as the food or the propellant of livestock.) as a raw material and fully drying this, capacity addition of the petroleum naphtha whose boiling point is 95-105 degrees C is carried out about 10 times, it is boiled, is filtered, and it separates into a part (extract) for a solid content (residue) and a liquid. Subsequently, the solid-like residue is obtained by carrying out distillation recovery of the naphtha from this extract.
[0004] This residue carries out a black tar a ** exception by filtering at about 75 degrees C, after adding and heating organic solvents, such as ethanol, an isopropanol, an acetone, and butyl alcohol, further and making it melt, since the tars other than a wax, such as a part for oil, a pitch, and a pitch, are contained. Cool the filtrate obtained in this way to near ordinary temperature, a part for a wax is made to separate, it filters, and a part for a wax is extracted. In addition, most organic solvents to use are collected by distilling filtrate.
[0005] It is difficult to industrialize on a large scale until it has been carried out by such technique, it has the following troubles in a manufacture of a ** and a ****** wax and it continues till present. That is, in a conventional method, although naphtha is cheap as (1) extracting solvent, there is concern in respect of safety. (2) Although hydrophilic organic solvents, such as a lower alcohol, are required for the naphtha extract in order to refine this, including so much impurities other than the wax part made into the purpose, the combined use with such a solvent and naphtha not only ****-izes a manufacturing facility and equipment, but it becomes the cause which raises a manufacturing cost. Therefore, the effective means was not found out although the development of the technique of performing extraction refining by the desirable high single solvent of safety had been desired.
[0006] Although it can consider using it with a lower alcohol as such an extracting solvent, in order to raise the extraction efficiency of a lipid system component like a wax, in the case of a lower alcohol, you have to use it as a solvent for a low water flow extremely. however, in what is generated by ** used as a raw material, and float ******, for example, a sugar pure process Usually, a colander is not obtained, if an actual extraction process is at operation by the water system, since about 70 - 80% of moisture is included. Simultaneously with impurities other than a wax (a part for oil, a pitch, pitch, etc.), by such system, the extractability of the wax part [itself] was also difficult to fall greatly and to raise an extraction efficiency called the purity and yield of the specified substance as a result. Moreover, in order to remove the moisture of a raw material, and the moisture in a recovery solvent, a great facility and a great cost are needed.
[0007]
[Problem(s) to be Solved by the Invention] Therefore, the purpose of this invention uses ** and float ****** as a raw material, and it is in offering the technique of extracting quality ** and a ****** wax constituent efficiently, using the high single solvent of safety.
[0008]

[Means for Solving the Problem] In order to solve the above-mentioned technical probrem, this invention persons found out zealously that the good lack of a quality and a ****** wax constituent were obtained by the high yield by cooling this extract and making a part for a wax separate after extraction processing in the state of an elevated temperature and pressurization using water alcohol as a result of the research. this invention is completed based on such knowledge. That is, the summary of this invention adds ethanol so that alcoholic concentration may become 50 % of the weight or more at the residue obtained from ** and ****** at the squeezing process at the time of manufacturing sugar, and after heating it under pressurization more than the boiling point of ethanol, it carries out a solid content a ** exception at the time of heat, and it is in the extraction method of ** characterized by making a crystal object separate from filtrate, and a ****** wax constituent.

[0009] The raw material used in this invention is remainder acquired at the squeezing process after it when squeezing processing of ** and the ****** is carried out in the process which manufactures sugar from ** and ****** by well-known technique, and the filter cake generated as such a residue at ****** at the time of squeezing of ** and ****** and the pure process of sugar is raised. Although these residues are obtained as a hydrated compound and can usually be applied by this invention also with a hydrated compound, it is desirable to make it dry from the point of workability and a manufacturing cost.

[0010] Ethanol is used as an extracting solvent, and it adjusts so that the alcoholic concentration in the system when adding in the aforementioned raw material may become 50 % of the weight or more. At less than 50 % of the weight, the extractability of the wax component made into the purpose falls. Therefore, the ethanol of various water contents can be used according to the moisture content of a raw material, and it is not necessary to necessarily use the non-water ethanol of a high grade as ethanol, in this invention. It is desirable to use water ethanol rather from recovery of an extracting solvent and the point of reuse, and the ethanol concentration is 70 - 90 % of the weight preferably 55% of the weight or more. It becomes impossible it to be unable to become difficult substantially to set up the alcoholic concentration in the aforementioned system to 50% of the weight or more, and to extract the target wax component efficiently at less than 55 % of the weight.

[0011] Next, in this invention, the mixture of the aforementioned raw material and ethanol is heated in the state of pressurization more than the boiling point of ethanol. the grade of pressurization -- 2kg/cm2 the above -- desirable -- 3-5kg/cm2 it is -- 2kg/cm2 the following -- the extractability of the specified substance -- low -- reverse -- 5kg/cm2 Even if it exceeds and pressurizes, the further extraction efficiency cannot be desired. Moreover, heating is preferably performed at 100-150 degrees C more than the boiling point of ethanol. Even if the purity of an extractability and an extract is low and exceeds 150 degrees C in heating of under the boiling point of ethanol, there are no effect and great difference of this invention. In such pressurization and the heating status, it stirs desirably, the contact to a raw material and ethanol is raised, and extraction processing is performed for 1 to 4 hours.

[0012] At the time of heat, the mixture obtained in this way is filtered by the technique that it is desirable and is well-known at 75-85 degrees C, and is divided into a solid content and filtrate. If it becomes lower than 75 degrees C at this time, the specified substance will separate, and it is removed by filtration processing, a fall of yield is imitated, and it is **. Subsequently, since filtrate is cooled below to a room temperature (25 degrees C) grade and yellow or a white crystal object separates at this time, ** and the ****** wax constituent which are made into the purpose can be obtained by carrying out this a ** exception. In addition, if simple distillation processing of the filtrate is collected and carried out, since it can refine as water ethanol whose ethanol concentration is about 90%, this is reusable as an extracting solvent.

[0013]

[Example] In the following examples and examples of a comparison, % is weight criteria.

100g (20% of water contents) of the dry matters of the filter cake which contains a part for a wax 4% obtained at the pure process of the juice honeydew of example 1 ** and ****** is taken in the pressure-proofing container with stirring equipment, ethanol 800ml is added 90%, and it is 2 3kg/cm. It pressurized and stirred at 120 degrees C for 2 hours. Subsequently, it filtered at 80 degrees C and separated into a solid content and filtrate. Filtrate was cooled to 25 degrees C, and after having put as it is for 5 hours and making a crystal separate, the crystal object was carried out the ** exception. They were the yield of 3.9g of a crystal object, and 78-82 degrees C of the melting points. In addition, it became light yellow when depigmentation processing of this crystal object was carried out in hydrogen peroxide solution 35 more%.

[0014] It carried out using the same equipment and the same raw material as example 2 example 1. That is, ethanol 1500ml is added to xeransis filter-cake (10% of water contents) 100g 70%, and it is 2 4kg/cm. It pressurized and stirred at 140 degrees C for 2 hours. It filtered at 80 degrees C after that, and separated into a solid content and filtrate. Filtrate was put at 25 degrees C for 6 hours, and the separated crystal object was carried out the ** exception. The yield of a

crystal object was 3.9g and 78-82 degrees C of the melting points.
[0015] It extracted, without pressurizing using the same equipment and the same raw material as example of comparison 1 example 1. Namely, ethanol 800ml was added to xeransis filter-cake (20% of water contents) 100g 90%, and it stirred at 75 degrees C by the ordinary pressure for 2 hours. Subsequently, it filtered at 80 degrees C and separated into a solid content and filtrate. Filtrate was cooled to 25 degrees C and the crystal object which put as it was and was separated for 5 hours was carried out the ** exception. There was little yield of a crystal object as 1g, and it was 70-75 degrees C of the melting points.
[0016] It extracted without heating using the same equipment and the same raw material as example of comparison 2 example 1. That is, ethanol 800ml is added to xeransis filter-cake (20% of water contents) 100g 90%, and it is 2 with nitrogen. It pressurized and stirred at 25 degrees C for 2 hours. Then, it filtered at 25 degrees C and separated into a solid content and filtrate. The crystal object was not separated although filtrate was put at 25 degrees C for 5 hours.
[0017] In technique given in example 3 example 1, the raw material was reset to the half-dry matter (40% of water contents) of this filter cake, 90% ethanol 2000ml which used for extraction processing at once, carried out simple distillation processing, and was collected was used, and also it was made these conditions and processed.
they were the yield of 3.8g of a crystal object, and 78-82 degrees C of the melting points.
[0018] In technique given in example 4 example 1, the raw material was reset in the split object (a part for 5% of water contents, and a wax : 0.2%) of the squeezing residue of ** and ******, ethanol 2000ml was used 90%, and also it processed on these conditions. The yield of the obtained crystal object was 0.18g and 78-82 degrees C of the melting points.
[0019]
[Effect of the Invention] According to this invention, the cheap residue of ** and float ****** is used as a raw material, and only using the high ethanol of the safety used also for food, in spite of being the extract operation in a water system, ** and the ****** wax constituent of a high grade can be extracted with high yield. Moreover, since extracting solvents are distilled and collected simply and this method can reuse them, it is excellent in economical efficiency.

[Translation done.]